

CLAIMS

1. A process for the production of amorphous and/or crystalline (mixed) oxides of metals, more particularly metals of the third to fifth main group or the secondary groups of the periodic system, with mean particle diameters
5 of the (mixed) oxides in the range from 1 to 500 nm and more particularly 1 to 100 nm, characterized in that, in an electrolysis cell equipped with a cathode and an anode, ions dissolved in organic electrolyte of that metal or those metals of which the (mixed) oxide is to be produced are electrochemically reduced at the cathode in the presence of an oxidizing
10 agent, characterized in that the cathode compartment (cathode half-cell) and the anode compartment (anode half cell) are separated from one another by a porous partition or separating wall (diaphragm) which allows current to pass through, i.e. is permeable to cations and anions, but impedes and in particular prevents the passage of elemental halogen from
15 the anode to the cathode compartment.
2. A process as claimed in claim 1, characterized in that the porous partition or separating wall in particular has a high halogen retention capacity.
3. A process as claimed in claim 1 or 2, characterized in that the
20 porous partition or separating wall is a porous glass frit, more particularly with a pore diameter of about 1 to 4 μm .
4. A process as claimed in claim 1 or 2, characterized in that the porous partition or separation wall is a polytetrafluoroethylene filter or a polytetrafluoroethylene membrane or a porous polyethylene film or
25 polyethylene disk, more particularly with a pore diameter of about 1 to 4 μm .
5. A process as claimed in claim 1 or 2, characterized in that the porous partition or separation wall is a proton-conducting membrane, preferably with a pore diameter of about 1 to 4 μm .
- 30 6. A process as claimed in claim 5, characterized in that the proton-

conducting ion exchanger membrane is in particular a perfluorinated ion exchanger membrane, preferably with a pore diameter of about 1 to 4 μm .

7. A process as claimed in any of claims 1 to 6, characterized in that organic electrolyte used is a substance which is liquid at temperatures in
5 the range from about -78°C to about $+260^{\circ}\text{C}$ and more particularly in the range from about 0°C to about 60°C at normal pressure and is preferably selected from the group consisting of ketones, alcohols and polyalcohols, ethers, nitriles and aromatic compounds.

8. A process as claimed in claim 7, characterized in that the organic
10 electrolyte is selected from the group consisting of n-propanol, i-propanol, glycerol and mixtures thereof with acetone, tetrahydrofuran, acetonitrile and toluene.

9. A process as claimed in any of claims 1 to 8, characterized in that the organic electrolyte additionally contains small quantities of water, more
15 particularly about 0.01 to about 2% by weight and preferably about 0.05 to about 1% by weight, based on the total quantity of the organic electrolyte and water.

10. A process as claimed in any of claims 1 to 9, characterized in that the organic electrolyte additionally contains a dissolved supporting
20 electrolyte which may be selected in particular from the group consisting of electrolyte-soluble hexafluorophosphates, sulfonates, acetyl acetonates, carboxylates and quaternary phosphonium and/or ammonium salts, more particularly with organic groups at the phosphorus and/or nitrogen, the supporting electrolyte being used together with surfactants which stabilize
25 the metal oxide particles produced and preferably being selected from the entire class of surfactants, the following compounds being particularly preferred: fatty alcohols, fatty alcohol ethoxylates, polyoxyethylene carboxylic acids and/or fatty acid ethoxylates.

11. A process as claimed in claim 10, characterized in that the
30 supporting electrolyte used is a quaternary ammonium compound which

may optionally carry one or more aryl and/or alkyl groups at the nitrogen and which is preferably present as a halide, such as tetrabutyl ammonium bromide.

12. A process as claimed in any of claims 1 to 11, characterized in that
5 the organic electrolyte has a temperature of 30 to 120°C.

13. A process as claimed in one or more of claims 1 to 12, characterized in that the oxidizing agent is selected from the group consisting of oxygen, hydrogen peroxide, peroxy compounds and oxo anions of the halogens chlorine, bromine or iodine where the halogen in particular has an oxidation
10 number of +1 to +5.

14. A process as claimed in claim 13, characterized in that the oxidizing agent is air (atmospheric oxygen), the oxidizing agent preferably being supplied to the reaction system by introduction of air into the electrolyte solution.

15. A process as claimed in any of claims 1 to 14, characterized in that the electrical d.c. voltage between cathode and anode is preferably adjusted so that a current density of 0.05 to 10 mA/cm², based on the anode area, and preferably in the range from 1 to 5 mA/cm², is obtained.

16. A process as claimed in claim 15, characterized in that an electrical
20 d.c. voltage of 1 to 100 volts is applied between cathode and anode.

17. A process as claimed in any of claims 1 to 16, characterized in that the electrolyte is agitated, more particularly by stirring, introduction of a gas and/or ultrasonication.

18. A process as claimed in any of claims 1 to 17, characterized in that
25 the anode and/or cathode material is a material which is inert under the selected electrolysis conditions and which is selected in particular from the group consisting of platinum and other platinum metals, gold, niobium, tantalum, tungsten, graphite and glassy carbon.

19. A process as claimed in any of claims 1 to 18, characterized in that
30 the metal ions dissolved in the electrolyte are produced by dissolving in the

electrolyte a salt of the metal or salts of those metals whose oxide or mixed oxide is to be produced.

20. A process as claimed in claim 19, characterized in that a halide, nitrate, acetate, sulfonate, carboxylate or hexafluorophosphate is used as the metal salt.

21. A process as claimed in any of claims 1 to 20, characterized in that, in the production of mixed oxides of at least two metals, the metal ions dissolved in the electrolyte are produced by dissolving a salt of one metal in the electrolyte and using an anode containing the other metal as a sacrificial anode.

22. A process as claimed in any of claims 1 to 21, characterized in that the metal (mixed) oxide formed is separated from the electrolyte and dried.

23. A process as claimed in claim 22, characterized in that the dried metal (mixed) oxide is subsequently calcined, more particularly at a temperature in the range from about 300 to about 1200°C and preferably at a temperature in the range from about 400 to about 1,000°C, or subjected to a hydrothermal treatment.

24. The use of the process claimed in any of claims 1 to 23 for the production of amorphous and/or crystalline oxides and mixed oxides of metals of the third to fifth main group or the secondary groups of the periodic system, more particularly of titanium, zirconium, chromium, molybdenum, iron, cobalt, nickel, indium, tin, lead and/or aluminium.